

BCA IG Newsletter

August 2009

Notes from the Chair

The Industrial Group has had a busy and productive half-year, with a great contribution to the Spring meeting, and plans well-advanced for the Autumn meeting this year. In 2010 we hope to have a joint XRF/XRD meeting, and a Pharma meeting, on top of our sessions at the BCA Spring Meeting. Details of all these meetings will be found on the website, and please remember that we're always happy to receive suggestions for meeting topics. In February 2009, Judith Shackleton organised a meeting with the British Society for Strain Measurement, held at Manchester University. At this year's Spring Meeting the IG was very involved with several great sessions. Contd. p2

Last Paper Version of this Newsletter

This is the last edition of the Newsletter in which a paper copy is sent out to all members. Future editions will be by e-mail only. If you don't receive the electronic version of the Newsletter then we don't have a correct e-mail address for you. Please sign up online or e-mail Dave Taylor to get onto our e-mail list. djtaylor@lineone.net

Forthcoming Events

- 5th November 2009 The World of Glass, St.Helens, Merseyside
Autumn Meeting – including a Young Crystallographers Meeting
- 12th to 15th April 2010
BCA Spring Meeting 2010
University of Warwick
- 12th (XRF) and 13th (XRD) May 2010
British Geological Survey (BGS)
Meeting

Log these dates in your diary NOW!

XRF Newsletter 9 published electronically in Aug 2009. View a copy on the web.



Charity Registration Number: 284718

World Wide Web addresses:

BCA <http://www.crystallography.org.uk>

IG <http://ig.crystallography.org.uk/ig.htm>

NOTE: We have changed the above Industrial Group web server so you may need to update your browser links.

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Newsletter Sponsorship.

The Industrial Group would like to thank all the sponsors of their paper Newsletter distribution over the past few years.

Notes from the Chair – contd.

We had a full parallel strand on XRF, covering a multitude of areas: portable instruments and environmental applications, to name but two, and the IG Plenary was given by Nick Marsh on the subject of Environmental Applications of XRF and XRD. The other IG sessions in this year's programs comprised sessions on pharmaceutical topics and two joint sessions with the BACG. I'd like to thank all our speakers, the IG Committee, and Nick Blagden of Bradford University for this year's IG program. Looking forward to the rest of the year, Mark Farnworth is organising the IG Autumn meeting, to be held on Nov 5th, at the 'World of Glass' museum in St. Helens. There will be an opportunity to look around the museum, which contains a wealth of exhibits, including the renowned Pilkington glass collection.

Last, but not least, we have had recent elections to the Committee. I'd like to welcome Margaret West and Cheryl Doherty to the IG Committee. The corollary is that some old friends are now leaving the Committee, and so here's a fond 'farewell' and big 'thankyou' to Steve Norval and Alison Burke. I hope that you all have a healthy and fun Summer and look forward to seeing many of you at the Autumn meeting.

Anne Kavanagh – AstraZeneca

Industrial Group E-mail Mailing lists – Online registration.

We now maintain separate lists for XRF and XRD mailings so please register for BOTH if you want to be kept totally in the picture. The IG sends about six E-mail notices each year to anyone interested (You don't even need to be a BCA member!). These inform of Newsletter postings and the various meetings we organise each year.

You can now register for our E-mail lists online - follow the link from the IG home page. There is an opportunity to be removed from the list with each mailing.

Please sign up NOW!

EDITORIAL

Welcome to this edition of the BCA Industrial Group's Newsletter. Most of the Newsletter contains reports from the XRD and XRF sessions held at this year's BCA Spring Meeting at the University of Loughborough.

As ever, the quality of the report writing is excellent and I'd like to say thank you to all the contributors who have taken the time to put the reports together. I would like to remind you that unedited versions of all the reports covered in this Newsletter can be found on the group's web site

<http://ig.crystallography.org.uk/ig.htm>

The web site has moved to a new server so please update your links to the pages. Thanks goes to our web-master David Taylor for managing this transfer.

Our next event is our Autumn Meeting which will be held at the World of Glass, St. Helens. See page 12 for details. The theme is the Impact of Crystallography in an Industrial Environment. If you work in the Industrial Sector or in a University Department that supports the Industrial sector then please consider giving a presentation. There will be a number of Young Crystallographers present so this is a good opportunity for us to show them real-life applications of our science.

Newsletter and Web Site Content

We continually try to improve the content of both the Newsletter and web pages and would like to ask you to submit articles etc. Do you use web sites for your day to day work in XRD and XRF? If yes, then which sites do you visit, let us know and send us a paragraph about what you use the site for and why you like it.

That's it for now. Enjoy your Newsletter.

Mark Farnworth – Pilkington Group Limited

BCA Spring Meeting – XRD Reports

21st to 23rd April 2009, Loughborough

XRF / XRD Plenary - Environmental Applications and associated session



Speaker - Left to Right: David Taylor (chair), Nick Marsh

There is a lot of information in climate records, which can lead to predictions about what we might be doing to the climate. Oxygen has two reasonably abundant stable isotopes, ^{16}O and ^{18}O . Chemical reactions often discriminate between them, but differentiation is greater in biological systems. Early work on geological and related samples involved carbonates, because it is easy to extract the CO_2 . Unfortunately, not all marine and lake environments are conducive to retaining carbonate phases, so there are gaps in the record.

Some plankton such as diatoms and radiolarians use silica to produce their skeletal frameworks. These can be used to plug the gaps in the oxygen isotope record, and also gives us access to information from freshwater lakes. Analytically, the oxygen is harder to get at, often requiring the use of HF. The problem gets worse, through the contamination of biogenic silicate samples with a variety of mineral and/or rock fragments with markedly different oxygen isotope signatures.

Decontamination of these samples can be complicated. Acid digestion is risky; it is moderately safe for the removal of carbonates, but does tend to use water, which of course contains oxygen. Oxidation of organics is often useful. A range of physical techniques can be applied, but they tend to fail when there are particles inside a diatom. XRD can be used to identify the contaminants, but this has its limitations: volcanic ash is often glassy, and the diatom silica tends to be amorphous too. With SEM and EDAX, it is possible to use XRF identification of the chemical signatures of the contaminants, and then remove their effects by a chemical mass balance: diatoms are roughly 92 % SiO_2 , 2 % Al_2O_3 , 6 % H_2O , and little else.

Samples, as received in the lab, are often only 50 - 60 mg. The customers want as much of the periodic table as possible, plus loss on ignition. The order of priority is usually the loss on ignition, followed by high-quality determinations of the main elements. The first choice for the technique is fusion beads with a high flux/sample ratio: the usual flux is $\text{Li}_2\text{B}_4\text{O}_7$. It is normal to find traces of Pt and Au in the beads - the crucibles (95Pt - 5Au) are not totally insoluble in the flux! A catch-weight system is used, without matrix corrections as the dilutions are so high.

The results are generally good for the major elements, but a number of special techniques have to be used in some cases: Zr/Sr and Y/Rb overlaps have to be corrected for. Pb tends to give odd results which are not yet fully understood. Work is still very much in progress improving the techniques. Cu, Ni and Cr are affected by contributions from various bits of the spectrometer: this could be hard to circumvent. To sum up, with equipment which is modern in all respects, and reliable reference materials, it is possible to get good results.

David Beveridge, HARMAN technology Ltd

BCA Spring Meeting – XRD Reports

21st to 23rd April 2009, Loughborough

Crystallography in the Pharmaceutical pipeline



Speakers - Left to Right: Jeremy Cockcroft, Matthew Johnson (Chair), David England, Cheryl Doherty.

Crystallography in Drug Development Cheryl Doherty, Pfizer

Cheryl gave an excellent talk presenting two case studies illustrating two uses of crystallography in the pipeline. The first case study described how computational methods such as polymorph prediction and molecular dynamics can be used to influence a polymorph screen by focusing the experiments to engineer a desired drug form with the correct hydrogen bonding. The second case study utilised non-ambient humidity conditions to collect crystal structures of a variable hydrate to understand the mechanism by which the compound could dehydrate and rehydrate. The information gathered from these crystal structures and solid state NMR, identified disordering of the fluoro-phenyl group as a potential channel for solvent movement, flipping of the ring acting as a revolving door.

The Crystal Structure is the Gold Standard for Proving of the Structure of a Drug Substance. What can be achieved for the Drug Product?

David England, Sanofi-Aventis

David's talk focused on the drug product, an area of research which in the past has had little interaction with the crystallographer, but with the advances of computational modelling

software, computer hardware and imaging techniques that can probe to sub-micron levels, the molecular and the macroscales are merging. The first case study used mesoscale simulations to rationalize the behaviour of complex polymer-based drug formulations and help to understand changes which occur during stability studies. The modelling showed how the drug interacted within the formulation, self-assembling to form a shell of drug forms around the propylene oxide rich regions, which is driven by the hydrophobic nature of the drug. The second study looked at tablet blends formulated by wet granulation and how accurate mapping of the surfaces by methods such as Raman and TOF SIMS mass spectrometry can be used to define the location of components and the degree of mixing.

From the Industrial via the Academic laboratory to the Court Room: Ciclovirs - A Pharmaceutical PXRD Case Study Jeremy K Cockcroft, UCL.

Jeremy's talk covered the court room and the synchrotron, two different areas of pharmaceutical endeavour. He presented the case of Roche versus Ranbaxy over valganciclovir and his personal account of his involvement in the case: from testing powder samples under non-ambient conditions, through producing documents to the court, to the final court appearance. Ranbaxy argued that the amorphous form they manufactured did not infringe on the crystalline patent of Roche. Jeremy demonstrated by quantitative analysis of amorphous content of the samples by XRPD, that the samples were X-ray diffraction amorphous and therefore demonstrated the information disclosed in the Ranbaxy patent to be true. The case is still in the court.

Matthew Johnson, GSK

BCA Spring Meeting – XRD Reports

21st to 23rd April 2009, Loughborough

Understanding API Phase Transitions



Speakers - Left to Right: Brett Cooper (Chair), Russell Johnstone, Nick Blagden, Paolo Avalle

Thermal Properties of Tolbutamide and Paracetamol by Real-Time Variable Temperature Raman Microscopy Paolo Avalle, MSD.

In the first IG presentation of the morning Paolo Avalle really hit the theme of the conference for dynamic crystallography, by demonstrating how, by applying Raman spectroscopy in real time, you can gain some insightful information into what is really happening during an API Phase Transition. Paolo described how the individual peaks in the Raman pattern could be assigned to specific areas of the molecule. He then demonstrated how these peaks could be tracked in real time during the heating of samples of paracetamol or tolbutamide. Then during phase transitions, he demonstrated how these peaks shifted. By looking at the crystal structures of the molecules he could then gain information about how the molecules were reorganising during the polymorphic transitions. By the use of principal component analysis he could plot the phase transitions with temperature and determine how many distinct phases were present during the transitions.

Co-Crystals and Crystal Growth Nicholas Blagden, University of Bradford

The second presentation of the morning saw a change to the scheduled program. J.Y. Khoo was unable to attend the conference so Nicholas Blagden heroically stepped in at the last minute to take the slot. Nick managed to put together an excellent presentation in only a couple of hours and even managed to aim towards the conference and session themes too. Nick described some of the theories of co-crystals and crystal growth. Demonstrating how co-crystal pairs could be selected by how strong their intermolecular interactions are likely to be, in a crystal engineering approach. He then demonstrated how you could screen for co-crystal formation using the Kofler contact method. He showed an example video of co-crystal growth at the interface between isonicotinamide and benzoic acid by hot stage polarised microscopy. He also demonstrated how ternary phase diagrams could be applied to solvent based co-crystallisation studies.

Identification of Driving Forces in High Pressure Phase Transitions Using the PIXEL Method.

Russell Johnstone, University of Edinburgh

In the last talk of the session Russell Johnstone applied real pressure! Up to 10 GPa in fact by the use of a Merrill-Bassett Diamond Anvil Cell. Russell described how crystals could be subjected to very high pressures while collecting single crystal X-ray or neutron powder data. He then described the phase transition that occurs at high temperature for L-serine monohydrate, showing the changes that occur to the crystal structure and intermolecular bond lengths. He also demonstrated how the PIXEL method can be used to understand why pressure-induced phase transitions occur.

Brett Cooper, MSD

BCA Spring Meeting – XRD Reports
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'Monitoring Crystals, Crystallization and Transformations'

Chick Wilson opened the first session with an overview of 'crystallisation phase space' in which he posed the question: should we expect the unexpected? And answered: yes, particularly polymorphism. Chick explored the question of whether favourable solid state properties of molecular materials can be achieved by understanding how those properties are determined by crystal structure. To do this, the structural chemist attempts to understand and control intermolecular bonds, particularly hydrogen bonds, using crystallisation, crystallography, solid state analysis and quantum chemical calculations.

Paul Barnes looked back at how in-situ synchrotron X-Ray diffraction has been applied, to study both the formation and the performance of functional materials. Time-resolved powder diffraction has been used to study rapid changes taking place in response to changes in temperature, pressure and chemical conditions. TEDDI (Tomographic Energy Dispersive Diffraction Imaging) has allowed spatially-resolved information to be gained in systems such as crystallisation of zeolite A on a ceramic base, (in which the crystallisation occurred in an unexpected place: the underside of the platform!).

Robert Hammond described how crystallisation control techniques can be used to produce 'the right stuff' for the pharmaceutical industry: crystals of the desired polymorph, crystal habit and size distribution. Control of polymorph is an essential in production of pharmaceuticals, but crystal habit and particle size distribution can also be important, since they affect secondary processing, such as filtration and powder flow. Robert explained how inline monitoring of particle size, polymorph and supersaturation can be achieved.

Roger Davey gave the first talk in the second session, on work aimed at understanding how structure evolves during the nucleation process. He investigated the relationship between the structure in the liquid crystal phase of *p*-azoxyanisole (*p*AA), and the structure of the crystalline phase which nucleated from the liquid crystal on cooling. He found that one polymorph, Form 1, was always formed, and Form 3 was never formed directly from the liquid crystalline phase. Roger concluded that the structure of the crystalline phase is dictated by the ordering within the supersaturated liquid crystalline phase, but that an energy barrier to nucleation still exists, since crystallisation was not observed, even after several weeks, at temperatures above 80°C.

Andrew Fogg described the use of *in-situ* synchrotron XRPD to follow the extent of reactions with time, and hence obtain invaluable information, such as activation energies, reaction order and nucleation models. Andrew gave an overview of some of the systems which have been studied. These included layered double hydroxides used as ion exchange materials, in which the stacking sequence and the formation of intermediates was investigated, and lanthanide intercalation compounds in which the original two dimensional structure was seen to convert to a three-dimensional structure, this observation being confirmed by high resolution electron microscopy.

The finale of the session was the announcement of the winner of the Industrial Group Prize for the best, industrially-relevant talk to be given in the Young Crystallographers Meeting. The prize was awarded to **Anne Stevenson from Bath University**, who described her work on novel metal-organic framework compounds. Anne received a cash prize and a bottle of bubbly from the IG Committee.

Anne Kavanagh, AstraZeneca.

BCA Spring Meeting XRD/XRF Joint Session – Environmental Applications Report - 21st to 23rd April 2009, Loughborough

XRD



Speakers - Left to Right: Richard Morris (chair), Peter Stacey, Didier Bonvin, Clive Roberts

Peter Stacey, Health and Safety Laboratory, treated us to a salutary reminder of the safety aspects of dealing with free silica. Outlining first what the HSE remit is including a proficiency testing scheme, forensic investigations, data preparation for court cases and environmental measurements.

Clive Roberts, University of Wolverhampton, described his study of industrial contamination in canals as a work in progress with As, Cd, Cr Pb, Hg & Se all being harmful to health. Lead was found to increase near to motorways. Many of the industries that previously lined the canals have contributed to specific contamination problems.

Didier Bonvin, Thermo Fisher Scientific, spoke to us about Environmental limits and outlined some specific applications. He spoke about the measurement of Sulphur in fuels and checking alternative fuels for contamination before allowing them to be burnt in for instance the lime industry. Another example was the screening of soils where old industrial sites are required to be turned safely into playgrounds. This limited the amount of soil for disposal and thereby trimmed the overall cost of such projects.

Martin Gill, Natural History Museum

XRF



Speakers - Left to Right: Richard Morris, Christine Vanhoof, Ian Croudace, Dave Taylor (chair)

Chris Vanhoof, Flemish Institute for Technological Research (VITO), spoke on How XRF fits into ROHS analysis. Directive 2002/95/EC restricts the use of certain hazardous materials in electrical and electronic equipment and this talk described the support given to the RoHS inspection campaign in Belgium.

Richard Morris, Morris Analytical X-ray Ltd, described his work to evaluate soil from excavated brown-field sites for use as top-soil. Concrete and metals were manually removed from mixed loads of up to 40 tons deposited for processing with the remainder screened using a 30 ton mechanical vibrator and electromagnet to generate coarse aggregate and the "top-soil".

Ian Croudace, National Oceanography Centre, who described his Itrax micro-XRF core scanner, just what the environmental sciences needed. This wondrous device is a multi-functional high-resolution XRF instrument that can measure element availability from Al to U with concentrations from trace major levels from step-sizes as small as 200 microns

Margaret West, West X-ray Solutions Ltd

BCA Spring Meeting – XRF Reports

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XRF Sessions – Foreword

Another successful three days of parallel XRF sessions were held at the 2009 Spring Meeting. We are indebted to all the speakers for providing presentations of a very high standard covering a vast array of topics. Thanks to the authors of the XRF reports spread over pages 7 -10.

Our next XRF meeting is on 12th May 2010 (with XRD on the 13th) so why not offer a talk; details are available on the XRF web pages. We need a new XRF logo – for details see issue 9 of our XRF logo on the web. If you are on our XRF mailing list you should have received a copy by E-mail, if not you need to register by filling out the form linked from our XRF home page.

Hand Held Applications Workshop



Speakers - Left to Right: Chris Calam, Mike Dobby, John Hurley, Debra Schofield, Richard Kilworth, Margaret West (chair), Steve Allott

The workshop began with an overview by the chair Margaret West of the use of hand held instruments. She covered the regulations involved in the safe working practice of these devices, the 1999 Ionising radiation regulations (mainly section 16), risk assessments and registration with the HSE. Also covered was the use of either a radioactive source or the now more common low wattage miniature x-ray tube typical of modern instruments.

Miniaturisation of the components for these devices over recent years has transformed the older "luggable" instruments into the modern truly hand held and portable devices. Silicon drift detectors (SDD) are now the detector of choice with their higher resolution and lower Z detection over the earlier PIN devices. They often work with a Personal digital assistant (PDA) running Windows mobile and bespoke software to cope with matrix effects, line overlaps, quantitative or qualitative analysis with pre-calibration available.

These small hand held devices allow *In situ* analysis away from the confines of the laboratory. The instrument is placed in contact with the sample and analytical results are immediately available to the operator allowing a value judgement on where to measure next.

With no sample preparation only the surface is measured with the bulk inaccessible and penetration limited by critical depth - a few microns for low Z elements and 1-2 mm for the K lines of high Z elements. The obvious problems from surface measurements are: patinas, contamination, weathering and moisture in soils. Applications include: Contaminated land, workplace monitoring, particulates on filters, surfaces and coatings, metal and alloy sorting, architectural buildings and monuments, geochemical prospecting, archaeological investigations, works of art and cultural heritage. Margaret's presentation was followed by live demonstrations from the "helping hands" from four hand held manufacturers in an area at the front of the lecture theatre that ensured that delegates were at least 2 metres from the working area. These were in order, Spectro, Oxford Instruments, Bruker AXS and Thermo Niton. The team and their instruments are shown in the above photograph. They covered a range of topics in their demonstrations including safe use, plastics, soils, metals and many more.

Dave Taylor, ICDD

BCA Spring Meeting – XRF Reports
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Portable Instruments



Speakers - Left to Right: Deborah Cane, Duncan Starke, Margaret West (chair), Phil Potts

Portable X-Ray Fluorescence Analysis - New Opportunities, New Challenges - Phil Potts "Open University"

Phil used a portable "luggable" X-Ray spectrometer (Spectrace TN9000), to analyse geochemical samples including a rock outcrop in the Preseli Mountains in North Wales. It was advantageous to use a portable X-Ray system to analyse this outcrop because conventional sampling is not permitted, as removal of rock material is illegal. Factors that affected the analysis were weathering and contamination of the rock samples with lichen and moss. These rocks were used to form the inner circle at Stonehenge.

A project he also worked on was determining the provenance of Neolithic stone axes found in various locations around the UK to determine which had originated from the Preseli Mountains in South Wales, as an outcome of his research half the axes found and previously attributed to the Preseli Mountains were determined to have originated elsewhere when non destructive X-Ray analysis was conducted.

Penguins & Precious Metals - the use of Portable XRF at Birmingham Museum & Art Gallery - Deborah Cane & Duncan Starke "Birmingham Museum & Art Gallery"

Deborah and Duncan gave a two-part presentation on the use of a Bruker portable XRF at Birmingham Museum & Art Gallery. First up was Deborah who uses the portable XRF to analyse samples from the natural history collection for the presence of toxic metals (incl. lead, arsenic and mercury) that may have been used in the taxidermy process. This is of particular importance because the exhibits maybe housed in an area where they can come into contact with visitors that may become contaminated with these toxic metals if exhibits are touched. Exhibits that contain these metals are only placed where they cannot come into contact with visitors.

Further work that Deborah carries out includes determining the provenance of artefacts including Egyptian bronze statues where simple non-destructive analysis helps determine fakes compared to the genuine artefacts.

Secondly Duncan gave a presentation on how he uses the portable XRF in administering the "Treasure Act 1996" to facilitate the rapid identification of an items composition, which has been found by people using metal detectors. This is of importance because historically any item that had been found required sending to the British Museum for formal identification, this was process is not quick and could lead to finder not being happy and they could work against the system. Examples of items he has checked include medieval annular brooches, Iron Age torcs and Roman coins.

Darren Musgrove, HSL

BCA Spring Meeting – XRF Reports

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New Developments in Instrumentation and TXRF



Speakers - Left to Right: Margaret West (chair), Christina Strelj, Malcolm Haigh, Armin Gross

Developments in TXRF Analysis -

Christina Strelj - TU Wien, Atominstitut der Österreichischen Universitäten.

A rapid overview of the applications of TXRF was presented, in particular the benefits of the technique, such as small required sample size. Techniques of increasing signal and optimising output were described, explaining how the technique allows the detector to be closer to the sample. A brief description was given of how to determine whether the point of interest was within or on the surface of a sample. This is done by using variations in the incident angle of x-rays. Further slides were used to present Christina's current research, particularly using synchrotron x-rays as a source, which gives distinct benefits over a standard x-ray tube. The talk finished with a summary of an advanced use of TXRF to map the location of contamination on silica wafers, whilst at the same time analysing whether sample compositions are within specification limits.

Trace element analysis of pharmacological, medical and biological samples by TXRF - Armin Gross - Bruker AXS Microanalysis GmbH

Armin gave a background to TXRF, and its application as a technique for analysis of liquids, powders, suspensions and thin films. Particular reference was made to the ability to analyse very small sample quantities through use of deposited thin films. These thin films give negligible matrix effects due to their lack of thickness, making quantification possible by using standard dopant elements without the need for further calibration. TXRF applied in this way can detect elements from Na-U. Discussion then continued with an introduction to the PicoFox bench-top TXRF. Armin highlighted the very short time to results achievable using this technique.

Energy dispersive XRF - its diversity and capabilities - Malcolm Haigh - Spectro Analytical UK Ltd.

Malcolm presented a series of slides to highlight the diversity of EDXRF as an analysis technique. Techniques, applications and typical uses of the main types of EDXRF, e.g. Low Resolution, Direct Excitation EDXRF were presented. These were linked to real situations and markets where the techniques were suitable.

An in-depth discussion of the benefits of polarised EDXRF was then presented, in which significant improvements in background noise and trace element detection were presented. The talk finished with a brief overview of the applications of Micro-XRF, including capillary optics, for use in situations such as RoSH analysis.

All three talks gave an excellent portrayal of recent advances in EDXRF technology and its consequent application to novel and interesting scientific challenges. The ability to analyse new materials, both through sampling and in-situ presents an exciting future for XRF

**Daniel Capon/Henry Foxhall,
Glass Technology Services**

BCA Spring Meeting – XRF Reports

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General Applications



Speakers - Left to Right: Yoshiyuki Kataoka, Simon FitzGerald, David Beveridge (chair), Stephen Davies.

Stephen Davies, PANalytical, discussed sources and causes of instrument drift - such as tubes, detectors and crystals. By far the largest cause of drift is the x-ray tube which deteriorates over time as tungsten coats the inside of the beryllium window. This effect is accentuated at high powers and a loss of intensity results in poor analytical precision. **Simon FitzGerald**, HORIBA Jobin Yvon Ltd, talked about new systems which now make mapping with Micro-XRF possible. A parallel x-ray beam is vital for this and 3 types are available. An aperture is a low cost option with low intensities, a mono-cap gives higher intensities at medium cost and a poly cap is best of all for intensities but is the most expensive.

Yoshiyuki Kataoka, Rigaku, Japan discussed micro spot technology in a WD-XRF. It yields high intensities, including light element performance, from a spot size of 100 microns. The spectrometer has a 4KW rhodium tube, sample masks, 10 crystals, 2 cameras, 2 detectors and the polycap system. The unit can be optimised for heavy elements with 50-80 micron spatial resolution.

Andy Scothern, Saint-Gobain Gyproc

Method Validation



Speakers - Left to Right: Ros Schwarz (chair), Debra Schofield, Eddie Birch, David Lowe, Paul Thomas.

David Lowe, United Kingdom Accreditation Service (UKAS) gave the Keynote lecture on Accreditation and Method Validation. Accreditation standards are all based on ISO9001 and IS17025 is used for laboratory accreditation. **Debra Schofield**, Oxford Instruments Analytical Ltd. described the ASTM process. Producing a new ASTM method is a 2 year process and is driven by people with sufficient time and money. A draft method is submitted to ASTM that shows a need for a technological breakthrough. **Eddie Birch**, CIQ Audit talked about ISO17025 accreditation. Advantages of accreditation include insurance claims, legal defence, efficiency and training. Carrying out a 'gap analysis' is a good plan for accreditation. **Prof. Paul Thomas**, University of Loughborough described Statistical Analysis and associated work flows in Quantitative Measurement Operations. All data should be presented with rigour and pride and this is the basis of a 100 hour post graduate program.

There is insufficient room in the Newsletter to include the XRF Trace Analysis report. Please view the report on the Industrial Group's Web Site:
<http://ig.crystallography.org.uk/ig.htm>

Forthcoming Meetings

Autumn Meeting 5th November 2009 The World of Glass, St. Helens, Merseyside

Impact of Crystallography in an Industrial Environment

The theme is the Impact of Crystallography in an Industrial Environment. The following talks have been submitted:

Preparation, Characterisation and Prediction of Physical Stability of Amorphous Materials during Pharmaceutical Development: Pair-Wise Distribution Function Helen Blade, AstraZeneca.

Applications of Crystallography in the Aerospace Industry Judith Shackleton, Materials Science Centre, University of Manchester

“XRD N’ Chips - What makes good semiconductor devices” Chris Staddon Senior Experimental Officer, School of Physics and Astronomy, University of Nottingham.

Powder X-ray Diffraction for Process and Product Support in Shell Global Solutions. Graham C. Smith, Shell Global Solutions UK.

XRD in the Imaging Industry
David Beveridge, Harman Technology
See web site for map and how to get there by train or car.

Call for Papers - Please call John Kaniuka Tel: 01695 54303 (Young Crystallographers) or Mark Farnworth with offers of talks at this meeting.

Organiser:
Mark Farnworth
Tel: 01695 54639
Email: Mark.Farnworth@pilkington.com

BCA Spring Meeting 2010 University of Warwick

Monday 12 April - Thursday, 15 April

The 2010 Spring Meeting will follow a theme of "Data Matters". The thinking behind this is to have a broad ranging theme which can encompass every aspect of a crystallographic study, whilst promoting an interdisciplinary feel for the conference. As usual there will be a Young Crystallographers Meeting on Monday 12th April which will run on into the Tuesday morning. The Programme Chair for the meeting is Dr Simon Coles, from the University of Southampton and the Programme Committee were at Warwick on July 15th to agree the format and session titles.

See the BCA web-site for updated information.
<http://www.crystallography.org.uk>

XRF / XRD Meeting British Geological Survey Keyworth, 12th to 13th May 2010

Call for Papers: See the IG XRD and XRF web pages for details

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Please see the Industrial Group's web site for the contact details of other Committee members.

<http://ig.crystallography.org.uk/ig.htm>